



APPROACH OF REPEATABILITY, REPRODUCIBILITY AND COMPARISON WITH CLASSICAL CHEMISTRY IN NIR PET FOOD ANALYSIS OF HUMIDITY

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Abstract: The study was carried out in order to optimize the non-invasive Near Infrared (NIR) technology in order to use the equipment for purposes of Animal production mobile laboratory for metabolic and nutritional advices in "2 Hours Farm Visits Program in West Romania". A sample of 22 pet feeds (10 samples with dog feed and 12 samples with cat feed from several producers and brands) was analyzed by classical physical-chemistry method and by NIR three parameters was calculated: 1. the repeatability limit (r value = 0.410), internal reproducibility limits (R_i = 1.583). By comparison with classical chemistry laboratory, also the reproducibility limits) was calculated (R value = 1.865). Practically, for an Animal production auto laboratory needs the estimation of repeatability and reproducibility limits can be effectuated for each type of feed, for any species including pets feed, concentrates for ruminants or swine or forages like silage or hay for dairy cattle sector.

- **Introduction** The aim of the study is to estimate the precision of analyses (accuracy or fidelity) of humidity, in terms of repeatability and internal reproducibility of NIR results obtain by INFRAEXACT FOSS and reproducibility (comparison with classical chemistry results from other laboratory) in order to use the equipment for purposes of Animal production mobile laboratory for metabolic and nutritional advices in "2 Hours Farm Visits Program in West Romania" for low input farms *

- **Material and method**

- *The material (subject) of analyses* was the pet feeds, from a Pets Experimental Unit a sample of 22 pet feeds (10 samples of dog feed and 12 samples of cat feeds, from several producers and brands) was extracted during one month period in order to measuring humidity (and other constituents such as: proteins, fat, NDF, cellulose etc) using classical chemistry and NIR analysis. Analysis results from NIR equipment comes from two spectra, and the average was considered value of measurement.



NIR Foss InfraXact®

- The first method was classical physical, performed in the Physical-chemical analysis for Soil – Plant – Fertilizers and Water laboratory "OSPA - USVT", and was used to evaluate the humidity through standardized method. The second analysis procedure was near infrared Near-infrared reflectance spectroscopy (NIR) with equipment Foss InfraXact® (Hillerød 3400, DK) from Auto Laboratory of Animal Production. The Foss InfraXact specters were developed by equipment producers' calibrations. The method is based on near-infrared (NIR) transmission spectroscopy, a secondary, correlative technique to predict the concentration of various constituents in biological or organic samples. The method was recently established by ISO 12099:2017 for application of near infrared spectrometry and by AOAC Official Method 2007.04 (Fat, Moisture, and Protein in Meat and Meat Products) for FOSS Food Scan™ Near-Infrared Spectrophotometer.

- **Results and discussions**

- **The repeatability limits (r value)** was calculated by standard deviation of NIR measurements for 22 samples (each one measured 10 times). The maximum standard deviation was 0.148 (see table 1), and the calculate repeatability limit was r value = $1.96 \times \sqrt{2} \times 0.148$, respectively $r = 0.410$. Checking the differences between two successive measurements and comparing with r value, all measurements were **admitted**.

- **Internal reproducibility limits (R_i value)** was calculated by standard deviation of NIR measurements for 22 samples (each one measured two times, in 10 consecutive days). The maximum standard deviation was 0.571 (see table 1), and the calculate repeatability limit was R_i value = 2.77×0.571 , **$R_i = 1.583$** . The differences between two successive measurements were compared with R value – in this case 99.5% (197/198) of measurements were **admitted** using average. For one sample the standard deviation was $1.590 > 1.583$. In those situations the median between three measurements (spectra) have to be considered and the cases not admitted not exceed more than 5 %.

- **The reproducibility limit (R value)** was calculated standard deviation of NIR measurements for 22 samples (measured two times, in two laboratories by classical chemistry and NIR). The standard deviation of differences in-between laboratory results differences was 0.673, and the calculate repeatability limit was R value = 2.77×0.673 , **R value = 1.865**. After comparison with R value, 95.45% (21 from 22) samples measurements were **admitted** using average. For two, the median between three measurements (spectra) have to be considered.

- **Conclusions** At the level of Animal production auto laboratory for NIR equipment and for internal analysis can be estimated the *repeatability limits*, *internal reproducibility limits* and *reproducibility limits*. Estimated limits are useful in order to establish the accuracy of NIR analysis for each type of feed and for control charts.

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